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The Electrolytic Preparation of
Alkali & Alkali-Earth Amalgams;
With Additional Notes
On Their Properties

Chemical Engineering

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THE ELECTROLYTIC PREPARATION OF ALKALI AND
ALKALI-EARTH~~S~~ AMALGAMS; WITH
ADDITIONAL NOTES ON THEIR PROPERTIES

BY

HARVEY CHILDS BENNETT

THESIS FOR THE DEGREE OF BACHELOR OF SCIENCE

IN CHEMICAL ENGINEERING

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THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

Harvey Childs Bennett

ENTITLED *The Electrolytic Preparation of Alkali
and Alkali Earth ~~Iron~~ amalgams; with additional notes
on their Properties.*

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

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Amalgams of the alkali and alkali-earth metals have been prepared by the following methods:

(1) Directly from the Metals:- The free alkali and alkali-earth metals unite more or less readily with mercury to form the corresponding amalgams. By this method concentrated amalgams can be prepared, but there exist the disadvantages that the free metals are not always available in the ordinary laboratory, and that in some cases special methods must be resorted to in order to induce the combination to take place.

(2) The Action of an Amalgam on a Salt Solution:- In general, one alkali or alkali-earth amalgam may be made from another by the action of a solution of a suitable salt of the first metal on the amalgam of the second. Since the reaction is a reversible one, a large excess of the concentrated salt solution is usually employed, and the treatment is repeated with fresh portions of the solution until the conversion is complete.

(3) The Electrolysis of a complex Alkali-mercury Salt in Aqueous Solution:- This method, although of theoretical interest, is not a common one for the preparations of amalgams, and it is mentioned only for the sake of completeness.

(4) The Electrolysis of a Salt Solution with a Mercury Cathode:- The best known electrolytic method for the preparation of these amalgams is that of Kerp (Z. Anorg. Chem. 17, 300 (1898)). Its description follows: Pure mercury was caused to flow from the capillary end of a funnel tube, in a fine stream, through a saturated solution

of the chloride of the metal to be amalgamated. The mercury was made the cathode by contact with a platinum wire; the anode consisted of carbon rods, which dipped into the salt solution. The vessel containing the chloride solution terminated below in a syphon tube, so arranged that only a small surface of the amalgam formed by the electrolysis was exposed to the action of the solution, and through which the amalgam flowed into a receptacle, provided at the bottom with an outlet tube bearing a stop-cock, and connected with a dropping funnel, by means of which the amalgam could be transferred back to the capillary funnel. The liquid amalgam was caused to repeat the circuit of the apparatus until it could no longer be made to flow in a continuous stream through the salt solution or until the evolution of hydrogen on the thread-like cathode became too rapid. To prevent oxidation of the amalgam during the process, dry hydrogen was continuously passed over the amalgam in the capillary funnel, in the receiver, and in the dropping funnel.

As an example of the efficiency of this method, Kerp says that on electrolyzing a saturated solution of potassium chloride for 4-5 hours at 8-10 volts, with 1000 grams of mercury as the cathode, he obtained 200 grams of solid amalgam, and that the liquid amalgam repeated the circuit of the apparatus 150 times. In the case of the lithium amalgam, the electrolysis lasted 4-5 hours at 14-16 volts, with 1000 grams of mercury as the cathode.

The liquid amalgam repeated the circuit about 100 times, and the yield was 40-50 grams of solid amalgam. It is evident that Kerp's method requires considerable time and labor, and it has the additional disadvantage, that it necessitates the use of special, rather complicated apparatus. Furthermore, the yields of solid amalgam leave much to be desired.

We have therefore sought to obtain better yields of solid amalgams in a simpler and less laborious manner. The efficiency of our method may be judged by comparing our yields of potassium and lithium amalgams with those of Kerp. With 250 grams of mercury as the cathode, a saturated solution of potassium chloride was electrolyzed for 50 minutes at 6 volts and 3.1-3.2 amperes: 113 grams of solid amalgam were obtained. With 250 grams of mercury as the cathode, a saturated solution of lithium chloride was electrolyzed for 90 minutes at 8.75-7 volts and 2.8-4 amperes; 71 grams of solid amalgam were obtained.

The method which we have employed is as follows: A convenient amount of mercury (200-250 grams) in a 150 cc. beaker was covered with about 50 cc. of the solution of an alkali or an alkali-earth chloride. The mercury was made the cathode by contact with a platinum wire, fused through the end of a glass tube which was partially filled with mercury and into which dipped the negative wire of the circuit. The anode was a piece of heavy platinum foil (2.1 cm. by 7.5 cm.), bent at ^aright angle, with one end

fastened electrically to the positive terminal of the circuit; all brass and copper connections were coated with paraffin in order to prevent contamination of the solution during electrolysis. This anode was dipped into the solution so that the free end was parallel with, and about 0.5 cm. below the surface of the salt solution. As considerable chlorine was evolved, the electrolysis was carried out in a hood. Six storage cells usually furnished enough current to allow the electrolysis to proceed satisfactorily. By means of standard Weston Instruments, the electromotive force and current strength could be read at will throughout the experiment.

Shortly after starting the electrolysis, crystals of solid amalgam began to appear on the surface of the cathode. These prevented the access of the metal deposited to the uncombined mercury underneath, and thus lowered the current efficiency. In order to obviate this difficulty, it was necessary to keep the cathode surface free from crystals, either by giving to the beaker a rotary, rocking movement, or by pushing the crystals below the surface of the liquid amalgam with a flattened glass rod bent at a right angle.

According to our experience, it is necessary to employ very pure salts, since traces of certain impurities such as iron and aluminium, greatly accelerate the interaction between the amalgam and the salt solution.

The electrolysis was discontinued in each case

when the volume of the hydrogen given off at the cathode was approximately equal to that of the chlorine evolved at the anode. At this point the salt solution was decanted, and the amalgam thoroughly washed with cold distilled water. It was then quickly dried with filter paper, representative samples were taken for analysis, and the remainder was preserved in small, glass stoppered weighing bottles.

The amalgams were analysed as follows:- The weighed samples were decomposed by means of tenth normal hydrochloric acid in excess, and the excess titrated back with tenth normal sodium hydroxide, with methyl orange as an indicator. From the amount of acid that was necessary to combine with the amalgamated metal in the sample, the quantity of the latter could be readily calculated.

The amalgams were allowed to stand quietly for several days, for the attainment of equilibrium between the liquid and solid phases; the percentages of solid and liquid were then determined by weighing the two phases, separated by filtration through chamois skin, in Gooch crucibles, on the suction pump.

It was found, while trying to verify some of the results obtained by Kerp and Bottger (Z. Anorg. Chem. 25, 1 (1900)), that the above method could not be relied upon for a complete separation of the liquid from the solid amalgams, and after some experimenting, the following procedure was decided upon. The solid amalgams, separated as far as possible from mother liquor by filtration through

chamois, were placed in glass tubes (about 20 cm. in length and with an internal diameter of 8 mm.). The amalgams were then melted by heating over a Bunsen flame, and the lower ends of the tubes containing the melted amalgams were tapped with considerable force on the desk in order to jar the amalgams, in compact form, into the lower part,—the traces of moisture, oxide, etc. rising to the upper surface. The tubes were then sealed off just above the upper surface of the amalgam and allowed to cool. In this way, an oxide free, compact, solid amalgam was obtained. By breaking the tubes into 2 cm. lengths, the amalgams could generally be pushed out in cylindrical form by means of a small glass rod; in some cases, however, it was necessary to crack the glass by means of a small hammer. The solid amalgams were then placed in open Gooch crucibles, and rotated at high speed in an electric centrifuge as long as mother-liquor continued to separate. As a result of this treatment, hard, compact cylinders of crystalline amalgam were in many instances obtained. These were freed from a superficial layer of oxide by means of a knife, and the bright crystals were immediately analysed.

Caesium Amalgam.

With 100 grams of mercury as the cathode, a solution of 13 grams of caesium chloride in 50 cc. of water was electrolyzed for 50 minutes at 6.5 - 7.0 volts and 3.0 - 2.5 amperes; the end temperature was 66°. The resulting product, even after washing with cold water, was quite liquid. On accidentally jarring the amalgam, after a few minutes, large crystals suddenly made their appearance at the surface, and on gentle stirring, the mass became semi-solid, owing to the separation of crystals. Continued stirring caused it to again liquify.

The amalgam was allowed to stand quietly for several days, after which it was filtered through chamois at 17°. It consisted of 72% solid and 28% liquid amalgam; the two phases analysed as follows:

Crystals-

8.2610 gr. $\Rightarrow$ 28.6 cc. N/10 HCl = 4.60% Cs.

6.7285 gr. $\Rightarrow$ 23.9 cc. N/10 HCl = 4.72% Cs.

5.2710 gr. $\Rightarrow$ 17.7 cc. N/10 HCl = 4.42% CS.

Mean = 4.60% Cs.

Liquid, saturated at 17°-

15.5370 gr. $\Rightarrow$ 32.0 cc. N/10 HCl = 2.74% Cs.

13.8400 gr. $\Rightarrow$ 28.7 cc. N/10 HCl = 2.76% Cs.

The calculated average caesium content was therefore 4.08%, indicating a current efficiency of 50%. When the saturated liquid mentioned above was cooled to 13°, crystals separated;

after standing for 6 hours at 18° , the remaining crystals were filtered off, and the liquid analysed:

11.6110 gr. $\rightleftharpoons$ 22.9 cc. N/10 HCl = 2.62% Cs.

14.7160 gr. $\rightleftharpoons$ 28.7 cc. N/10 HCl = 2.60% Cs.

These figures are thought to be more correct for the solubility of caesium in mercury than those obtained at 17° , since in the former case, the solution was probably slightly supersaturated.

A saturated solution of caesium in mercury was allowed to stand in cracked ice for 3 hours. The liquid amalgam was then filtered off from the large, well formed crystals which had separated, in a previously cooled Gooch crucible. It was then at once analysed:

Liquid, saturated at 0° , (stood 3 hours in ice)

22.3990 gr. $\rightleftharpoons$ 33.4 cc. N/10 HCl = 1.98% Cs.

18.0790 gr. $\rightleftharpoons$ 27.0 cc. N/10 HCl = 1.98% Cs.

Liquid, saturated at 0° , (stood 5 hours in ice)

15.7772 gr. $\rightleftharpoons$ 23.3 cc. N/10 HCl = 1.96% Cs.

22.6529 gr. $\rightleftharpoons$ 33.4 cc. N/10 HCl = 1.95% Cs.

Crystals, separated at 0° ,

4.3850 gr. $\rightleftharpoons$ 15.1 cc. N/10 HCl = 4.58% Cs.

5.2920 gr. $\rightleftharpoons$ 17.9 cc. N/10 HCl = 4.50% Cs.

As the foregoing figures for the crystals do not correspond with any simple formula, the presence of mother-liquor in the crystals was suspected; The crystals were therefore placed in Gooch crucibles and rotated for several minutes in a large hand centrifuge. This caused the separation of

considerable mother-liquor; the analysis of the crystals follows:

5.2895 gr. $\rightleftharpoons$ 18.8 cc. N/10 HCl = 4.73% Cs.

4.7050 gr. $\rightleftharpoons$ 16.9 cc. N/10 HCl = 4.77% Cs.

Another sample of the crystalline amalgam, separated from mother liquor by means of the electric centrifuge, gave on analysis:

7.4671 gr. $\rightleftharpoons$ 28.4 cc. N/10 HCl = 5.05% Cs.

7.9636 gr. $\rightleftharpoons$ 30.4 cc. N/10 HCl = 5.07% Cs.

The formulae CsHg_{13} and CsHg_{12} require 4.86% Cs and 5.25% Cs, respectively.

Rubidium Amalgam.

With 100 grams of mercury as the cathode, a solution of 10 grams of rubidium chloride in 50 cc. of water was electrolyzed for 40 minutes at 8.0 volts and 2.25 amperes; the end temperature was 54°. Crystals of amalgam appeared on the surface of the cathode 13 minutes after the electrolysis was started, and continued to form rapidly until the end. At the end temperature of 54° the amalgam was pasty, but on washing with cold water it solidified to a crystalline mass, which could be picked up without the separation of liquid. The amalgam analysed as follows:

8.7287 gr. $\rightleftharpoons$ 29.3 cc. N/10 HCl = 2.87% Rb.

16.6210 gr. $\rightleftharpoons$ 51.2 cc. N/10 HCl = 2.63% Rb.

12.1770 gr. $\rightleftharpoons$ 39.4 cc. N/10 HCl = 2.77% Rb.

7.5676 gr. $\rightleftharpoons$ 26.9 cc. N/10 HCl = 3.04% Rb.

Mean = 2.88% Rb.

The current efficiency was therefore 60%.

The amalgam was allowed to stand quietly for several days, after which it was filtered through chamois at 19.5°. It consisted of 68% of solid and 32% of liquid. The two phases analysed as follows:

Crystals;

9.6600 gr. $\rightleftharpoons$ 40.6 cc. N/10 HCl = 3.59% Rb.

9.1620 gr. $\rightleftharpoons$ 37.9 cc. N/10 HCl = 3.54% Rb.

Liquid, saturated at 19.5°;

16.9770 gr. $\rightleftharpoons$ 24.3 cc. N/10 HCl = 1.23% Rb.

24.0720 gr. $\rightleftharpoons$ 33.0 cc. N/10 HCl = 1.20% Rb.

15.1000 gr. $\rightleftharpoons$ 21.5 cc. N/10 HCl = 1.22% Rb.

A saturated solution of rubidium amalgam was allowed to stand in cracked ice for 2 hours. The crystals which separated were filtered off in a previously cooled Gooch crucible, and rotated in a hand centrifuge until mother liquor no longer separated from them; these crystals gave on analysis:

6.5560 gr. $\rightleftharpoons$ 28.2 cc. N/10 HCl = 3.68% Rb.

3.4710 gr. $\rightleftharpoons$ 15.1 cc. N/10 HCl = 3.72% Rb.

The previously melted solid amalgam, separated from mother liquor by rotation in the electric centrifuge, gave on analysis:

2.1383 gr. $\rightleftharpoons$ 9.8 cc. N/10 HCl = 3.92% Rb.

2.5717 gr. $\rightleftharpoons$ 12.3 cc. N/10 HCl = 4.09% Rb.

1.8636 gr. $\rightleftharpoons$ 7.8 cc. N/10 HCl = 3.57% Rb.

1.6512 gr. $\rightleftharpoons$ 6.9 cc. N/10 HCl = 3.57% Rb.

The average of the last six analyses is 3.76% Rb, while the formula RbHg_{11} requires 3.74% Rb.

Potassium Amalgam.

With 250 grams of mercury as the cathode, 50 cc. of a saturated solution of potassium chloride were electrolyzed for 50 minutes at 6.0 volts and 3.1 - 3.2 amperes. Crystals appeared at the surface of the cathode 15 minutes after the electrolysis was begun; after 40 minutes the mass was paste-like in consistency. The final product, washed with cold water, was a solid cake of small, needle-like crystals.

The amalgam gave on analysis:

$$3.9777 \text{ gr. } \rightleftharpoons 12.2 \text{ cc. N/10 HCl} = 1.20\% \text{ K.}$$

$$5.5424 \text{ gr. } \rightleftharpoons 16.7 \text{ cc. N/10 HCl} = 1.19\% \text{ K.}$$

The current efficiency was therefore 79%.

The amalgam was allowed to stand quietly for several days, after which it was filtered through chamois at 20°. It consisted of 58% of solid and 42% of liquid. The two phases gave on analysis:

Crystals:

$$8.5470 \text{ gr. } \rightleftharpoons 37.1 \text{ cc. N/10 HCl} = 1.71\% \text{ K.}$$

Liquid, saturated at 20°:

$$29.2800 \text{ gr. } \rightleftharpoons 34.9 \text{ cc. N/10 HCl} = 0.47\% \text{ K.}$$

The same solid amalgam, freed from mother liquor by filtration through chamois, and subsequent rotation in the electric centrifuge, gave on analysis:

$$7.0071 \text{ gr. } \rightleftharpoons 32.1 \text{ cc. N/10 HCl} = 1.79\% \text{ K.}$$

$$6.7341 \text{ gr. } \rightleftharpoons 30.4 \text{ cc. N/10 HCl} = 1.77\% \text{ K.}$$

The previously melted solid amalgam, freed from mother-liquor by rotation in the electric centrifuge, gave

on analysis:

2.8310 gr. $\rightleftharpoons$ 12.3 cc. N/10 HCl = 1.70% K

3.2632 gr. $\rightleftharpoons$ 14.4 cc. N/10 HCl = 1.73% K

2.0641 gr. $\rightleftharpoons$ 9.9 cc. N/10 HCl = 1.87% K

2.3067 gr. $\rightleftharpoons$ 10.8 cc. N/10 HCl = 1.83% K

1.9049 gr. $\rightleftharpoons$ 8.8 cc. N/10 HCl = 1.81% K

2.6004 gr. $\rightleftharpoons$ 11.7 cc. N/10 HCl = 1.76% K

The mean composition of the crystalline samples is 1.77% K, while the formula KHg_{11} requires 1.75% K.

Sodium Amalgam.

With 250 grams of mercury as the cathode, 50 cc. of a saturated solution of sodium chloride were electrolyzed for 105 minutes at 6.0 volts and 3.0 amperes. 90 minutes after the electrolysis was begun the amalgam was paste-like in consistency; at the end of the electrolysis it was a solid cake of needle-like crystals. On analysis it gave:

11.8148 gr. $\rightleftharpoons$ 78.3 cc. N/10 HCl = 1.52% Na

13.6830 gr. $\rightleftharpoons$ 90.1 cc. N/10 HCl = 1.51% Na

The current efficiency was therefore 84%.

The amalgam was allowed to stand quietly for several days, after which it was filtered through chamois at 22°. It consisted 72% of solid and 28% of liquid. The two phases analysed as follows:

Crystals:

3.3093 gr. $\rightleftharpoons$ 30.4 cc. N/10 HCl = 2.12% Na

2.2957 gr. $\rightleftharpoons$ 21.6 cc. N/10 HCl = 2.17% Na

(13)

3.0184 gr. $\rightleftharpoons$ 25.8 cc. N/10 HCl = 1.97% Na

3.6030 gr. $\rightleftharpoons$ 31.6 cc. N/10 HCl = 2.02% Na

Liquid, saturated at 22°;

11.8148 gr. $\rightleftharpoons$ 78.3 cc. N/10 HCl = 1.52% Na

13.6830 gr. $\rightleftharpoons$ 90.1 cc. N/10 HCl = 1.51% Na

The previously melted solid amalgam, freed from mother-liquor by rotation in the electric centrifuge, gave on analysis:

2.3212 gr. $\rightleftharpoons$ 24.1 cc. N/10 HCl = 2.40% Na

2.4024 gr. $\rightleftharpoons$ 24.8 cc. N/10 HCl = 2.37% Na

(These samples were scraped as free as possible from oxide with a knife; on covering each with water, about 1.0 cc. of N/10 HCl was necessary to neutralize the free alkali)

2.2841 gr. $\rightleftharpoons$ 21.1 cc. N/10 HCl = 2.13% Na

2.5620 gr. $\rightleftharpoons$ 23.8 cc. N/10 HCl = 2.14% Na

(These samples treated as above, required 0.3 cc. N/10 HCl to neutralize the free alkali.)

The average of the last four analyses is 2.26% Na, while the formula NaHg_5 requires 2.25% Na.

Lithium Amalgam.

With 250 grams of mercury as the cathode, 50 cc. of a saturated solution of lithium chloride were electrolyzed for 90 minutes,-- the first 30 minutes at 7.0 volts and 1.5 amperes, and the last 60 minutes at 8.5 volts and 3.25 amperes; the end temperature was 70°. It was found advantageous to keep the surface of the cathode covered

with solid lithium chloride during the electrolysis: in all, 50 grams of lithium chloride were added to the original saturated solution. At the end of the electrolysis, the amalgam was a semi-solid mass of crystals. It analysed as follows:

17.4413 gr. $\rightleftharpoons$ 98.7 cc. N/10 HCl = .396% Li

14.6079 gr. $\rightleftharpoons$ 82.5 cc. N/10 HCl = .395% Li

The current efficiency was therefore 96%.

The amalgam was allowed to stand quietly for several days, after which it was filtered through chamouis at 22°. It consisted of 42% of solid and 58% of liquid. The two phases analysed as follows:

Crystals;

2.7610 gr. $\rightleftharpoons$ 36.2 cc. N/10 HCl = .896% Li

3.0131 gr. $\rightleftharpoons$ 37.0 cc. N/10 HCl = .863% Li

Liquid, saturated at 22°;

23.1987 gr. $\rightleftharpoons$ 15.6 cc. N/10 HCl = .047% Li

33.3040 gr. $\rightleftharpoons$ 22.3 cc. N/10 HCl = .047% Li

The previously melted solid amalgam, freed from mother-liquor by rotation in the electric centrifuge, gave on analysis:

1.1083 gr. $\rightleftharpoons$ 17.4 cc. N/10 HCl = 1.104% Li

1.0788 gr. $\rightleftharpoons$ 17.0 cc. N/10 HCl = 1.108% Li

1.0421 gr. $\rightleftharpoons$ 16.1 cc. N/10 HCl = 1.086% Li

1.3631 gr. $\rightleftharpoons$ 21.2 cc. N/10 HCl = 1.093% Li

1.5977 gr. $\rightleftharpoons$ 21.9 cc. N/10 HCl = .964% Li

(When covered with water, about 1.0 cc. N/10 HCl was necessary to neutralize the free alkali in each case)

The average of the last 5 analyses is 1.05% Li, while the formula of LiHg_3 requires 1.16% Li.

Barium Amalgam.

With 250 grams of mercury as the cathode, 50 cc. of a saturated solution of barium chloride were electrolyzed for 138 minutes at 6.0 - 7.0 volts and 2.1 - 1.75 amperes; the end temperature was 69° . Not much stirring of the amalgam was necessary in this case. At the end of the electrolysis the amalgam was a solid mass of crystals. The analysis gave:

9.5100 gr. $\Rightarrow$ 48.3 cc. N/10 HCl = 3.49% Ba

7.1000 gr. $\Rightarrow$ 36.0 cc. N/10 HCl = 3.48% Ba

The current efficiency was therefore 77%.

The amalgam was allowed to stand quietly for several days, after which it was filtered through chamois at the ordinary room temperature. It consisted of 65% of solid and 35% of liquid. The two phases analysed as follows:
Crystals;

16.8640 gr. $\Rightarrow$ 39.7 cc. N/10 HCl = 5.48% Ba

Liquid;

18.4580 gr. $\Rightarrow$ 9.8 cc. N/10 HCl = 0.73% Ba

The previously melted solid amalgam, separated from mother-liquor by rotation in the electric centrifuge, gave on analysis:

1.2542 gr. $\Rightarrow$ 10.1 cc. N/10 HCl = 5.53% Ba

1.3929 gr. $\Rightarrow$ 11.6 cc. N/10 HCl = 5.72% Ba

(When covered with water, 0.1 cc. N/10 HCl was required to neutralize the free alkali)

2.0701 gr. $\Rightarrow$ 17.5 cc. N/10 HCl = 5.81% Ba

1.3645 gr. $\Rightarrow$ 11.9 cc. N/10 HCl = 5.99% Ba

3.1711 gr. $\Rightarrow$ 23.7 cc. N/10 HCl = 5.13% Ba

(On covering sample with water, 0.6 cc. N/10 HCl was necessary to neutralize the free alkali)

2.8532 gr. $\Rightarrow$ 23.5 cc. N/10 HCl = 5.65% Ba

(1.2 cc. N/10 HCl were required to neutralize the free alkali).

The average of the last six analyses is 5.64% Ba, while the formula BaHg_{11} requires 5.88% Ba.

Strontium Amalgam.

With 260 grams of mercury as the cathode, 50 cc. of a saturated solution of strontium chloride were electrolyzed for 90 minutes at 8.0 - 7.0 volts and 2.5 - 2.75 amperes; the end temperature was 43°. Considerable hydrogen was evolved during the electrolysis. The presence of strontium chloride crystals on the surface of the cathode was found to be advantageous. At the end of the electrolysis, the amalgam was paste-like in consistency; it analysed as follows:

28.3378 gr. $\Rightarrow$ 128.0 cc. N/10 HCl = 1.99% Sr

14.0130 gr. $\Rightarrow$ 62.6 cc. N/10 HCl = 1.96% Sr

The current efficiency was therefore 80%.

The amalgam was allowed to stand quietly for several days, after which it was filtered through chamois skin at 23°.

It consisted of 48.1% of solid and 51.9% of liquid. The two phases gave on analysis;

Crystals;

9.2750 gr. $\rightleftharpoons$ 52.7 cc. N/10 HCl = 2.82% Sr

6.9155 gr. $\rightleftharpoons$ 44.3 cc. N/10 HCl = 2.80% Sr

Liquid, saturated at 23°;

17.5886 gr. $\rightleftharpoons$ 45.1 cc. N/10 HCl = 1.12% Sr

14.6585 gr. $\rightleftharpoons$ 37.6 cc. N/10 HCl = 1.13% Sr

The previously melted solid amalgam, separated from mother-liquor by rotation in the electric centrifuge, gave on analysis:

3.3934 gr. $\rightleftharpoons$ 28.4 cc. N/10 HCl = 3.66% Sr

(On covering with water, 1.1 cc. N/10 HCl were necessary to neutralize the free alkali)

2.0952 gr. $\rightleftharpoons$ 16.9 cc. N/10 HCl = 3.53% Sr

1.7613 gr. $\rightleftharpoons$ 14.0 cc. N/10 HCl = 3.48% Sr

(On covering the above two samples with water, 0.8 cc. N/10 HCl was necessary to neutralize the free alkali)

The average of the last three analyses is 3.56% Sr, while the formula SrHg_{11} requires 3.51% Sr.

Calcium Amalgam.

Owing to the fact that almost as fast as the calcium combined with the mercury, the amalgam was decomposed by the solution, it was not possible to prepare a concentrated amalgam of this metal; another difficulty was the formation of a mixture of black, finely divided mercury and calcium

hydroxide, which finally broke the circuit. Solutions of several different salts of calcium were tried as electrolytes but without success; calcium acetate solution gave the best results.

With 250 grams of mercury as the cathode, 35 cc. of a saturated solution of calcium acetate were electrolyzed for 50 minutes at 10.0 volts and 1.25 amperes; the end temperature was 75°. Twenty minutes after the electrolysis was begun, there appeared, around the sides of the beaker below the surface of the mercury, white calcium hydroxide together with some patches of black finely divided mercury. The final product, after washing, consisted of liquid amalgam, which analysed as follows:

28.9230 gr. $\rightleftharpoons$ 13.3 cc. N/10 HCl = 0.092% Ca

42.7520 gr. $\rightleftharpoons$ 19.4 cc. N/10 HCl = 0.091% Ca

Summary.

Without the use of complicated apparatus, and at comparatively low voltages, pure amalgams of the alkali and alkali-earth metals have been prepared, in less time and with better yields than ^{by} any previously described electrolytic process.





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